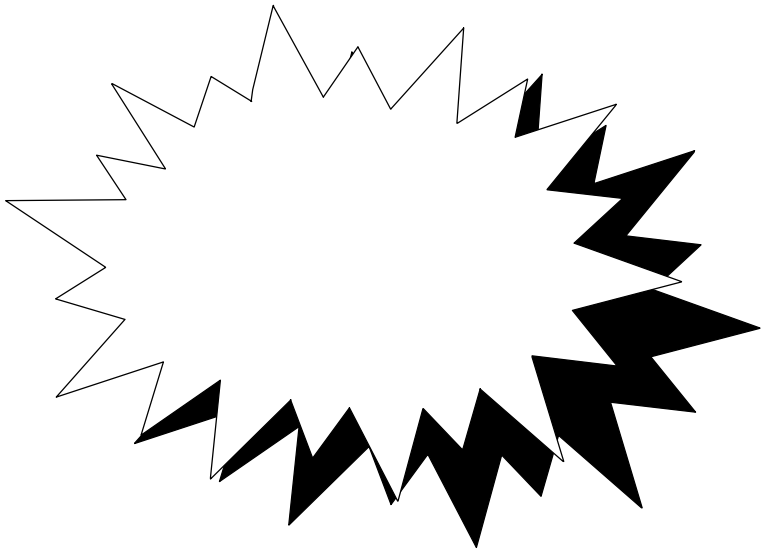

Chemical Analysis and Testing Task

Laboratory Analytical Procedure

LAP-012

Procedure Title: Standard Test Method for Moisture, Total Solids, and Total Dissolved Solids in Biomass Slurry and Liquid Process Samples

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Standard Test Method for Moisture, Total Solids, and Total Dissolved Solids in Biomass Slurry and Liquid Process Samples

Laboratory Analytical Procedure #012

1. Introduction

1.1 A critical component of evaluating processes involved in the conversion of biomass is the accurate compositional analysis of samples from those process streams. Although these process samples can be analyzed as a whole sample (slurry), often they are separated into solid and liquid fractions and analyzed separately. To be meaningful, the results of chemical analyses of biomass are typically reported on a dry weight basis. The following procedure describes the method used to determine the total solids (or moisture) content of a slurry or the liquid fraction of a biomass process sample. This procedure can also be used to determine the total dissolved solids content of such samples.

2. Scope

2.1 This test method covers the determination of total solids (or moisture) in slurries or the liquid fraction of samples generated during the pretreatment, fractionation, and fermentation of biomass.

3. References

3.1 Moore, W.E., and D.B. Johnson. 1967. *Procedures for the Chemical Analysis of Wood and Wood Products*. Madison, WI: USDA Forest Products Laboratory, U.S. Department of Agriculture.

3.2 Technical Association of the Pulp and Paper Industry (TAPPI) Standard Method T OS-63.

3.3 NREL Chemical Analysis and Testing Task Laboratory Analytical Procedure #001, "Standard Test Method for the Determination of Total Solids in Biomass".

4. Terminology

4.1 The total solids content of a sample is the amount of material left as a residue upon drying at 105^{o}C to constant weight.

4.2 The total dissolved solids content applies to liquid or slurry samples and is defined in

this procedure as the amount of residue from the filtrate of a 0.2 μm filtered sample that has been dried at 105°C to constant weight.

5. Significance and Use

5.1 The total solids content is a measure of the amount of solids suspended or dissolved in a process liquid or slurry. Conversely the moisture content is a measure of the amount of water (and other components volatilized at 105°C) present in such samples.

5.2 The results of chemical analyses of processed biomass samples are typically reported on a dry weight basis. The total solids content of a sample is used to convert the analytical results obtained on another basis to that of a dry weight basis.

6. Apparatus

6.1 Automatic infrared moisture analyzer (such as Denver Instrument Company IR-100 or equivalent) or a convection oven, with temperature control of 105 ± 2°C.

6.2 Analytical balance, sensitive to 0.1 mg.

6.3 Desiccator.

7. Reagents and Materials

7.1 Syringe filter, 0.8 μm prefilter over 0.2 μm final filter (such as Gelman Acrodisc μF or equivalent).

7.2 Aluminum foil weighing dishes.

7.3 Quartz pads for weighing dishes.

7.4 Transfer pipettes.

8. ES&H Considerations and Hazards

8.1 Follow all applicable NREL Laboratory Specific Hygiene Plan guidelines.

9. Sampling, Test Specimens and Test Units

9.1 Test specimens suitable for analysis by this procedure are slurries and the liquid fraction of samples generated during the pretreatment, fractionation, or fermentation of biomass. If the total solids (or moisture) contents of the solid fraction of these process samples are to be determined, the Laboratory Analytical Procedure #001, "Standard Method for the Determination of Total Solids in Biomass", must be used instead.

9.2 The test specimen shall consist of approximately 3 to 10 g of sample obtained in such a manner as to ensure that it is representative of the entire lot of material being tested. Thorough mixing of slurries and liquid fractions with precipitates is of particular importance.

10. Convection Oven Procedure

10.1 Accurately weigh a predried aluminum foil weighing dish to the nearest 0.1 mg and then tare the balance.

10.2 For a total solids or moisture determination, thoroughly mix the sample and then weigh out 3 to 10 g, to the nearest 0.1 mg, into the tared weighing dish. For total dissolved solids, the sample added to the tared weighing dish should first be passed through a 0.8/0.2 μm syringe filter.

10.3 Place the sample into a convection oven at 105 ± 2°C and dry to constant weight (±0.1% change in the amount of moisture present upon one hour of reheating). Typically overnight drying is required for very wet samples.

10.4 Remove the sample from the oven and place in a desiccator; cool to room temperature.

10.5 Weigh the dish containing the oven-dried sample to the nearest 0.1 mg.

11. Infrared Moisture Analyzer Procedure

11.1 Program the automatic moisture analyzer for an analysis temperature of 105°C and for a pre-determined end of analysis criteria of a rate of weight change that does not exceed 0.05% in one minute.

11.2 Verify that the instrument has reached the analysis temperature of 105°C and then place an aluminum foil weighing dish with quartz pad on the balance pan. Wait five minutes to ensure that the dish and pad are completely dry and then tare the balance.

11.3 For a total solids or moisture determination, quickly transfer 3 to 10 g of the thoroughly mixed sample to the quartz pad in the weighing dish. For total dissolved solids, the sample added to the quartz pad should first be passed through a 0.8/0.2 μm syringe filter.

11.4 As soon as the instrument balance produces a stable weight, proceed with the analysis.

11.5 Once the sample has been dried to constant weight, as determined by the programmed analysis parameters, the analysis will be automatically terminated by the instrument.

12. Calculations

12.1 Calculate the percent total solids or the percent moisture on a 105°C dry weight basis as follows (the automated moisture analyzer will provide the selected calculated value as part of the instrument printout):

$$\%\ Total\ solids = \frac{weight\ dried\ sample\ plus\ dish - weight\ dish}{weight\ sample\ as\ received} \times 100$$

$$\%\ Moisture = [1 - \frac{(weight\ dried\ sample\ plus\ dish - weight\ dish)}{weight\ sample\ as\ received}] \times 100$$

12.2 Calculate the percent total dissolved solids on a 105°C dry weight basis as follows (the automated moisture analyzer will provide the selected calculated value as part of the instrument printout):

$$\%\ Total\ dissolved\ solids = \frac{weight\ dried\ sample\ plus\ dish - weight\ dish}{weight\ sample\ after\ 0.8/0.2\ um\ \ filtration} \times 100$$

13. Report

13.1 Report the result as a percentage with two decimal places.

13.2 For replicate analyses of the same sample, report the average, standard deviation, and relative percentage difference.

14. Precision and Bias

14.1 The precision of this analysis is determined by evaluation of %RPD data from samples of many different types and assumes a 95% confidence interval. Based on this evaluation, the precision of the infrared moisture analyzer procedure is 3% using a Denver Instrument IR-100. The precision for the convection oven procedure is 1%.

14.2 An inherent error in any oven drying procedure is that volatile substances other than water are removed from the sample during drying.

15. Quality Control

15.1 *Reported significant figures:* All data is reported with two decimal places.

15.2 *Replicates:* All samples are run in duplicate.

15.3 *Relative percent difference criteria:* For the infrared drying method the maximum %RPD for duplicate analysis of a liquid samples is 9%. For the oven method the maximum %RPD is 3%. If the %RPD is exceeded, the sample should be rerun.

15.4 *Blank:* This gravimetric analysis utilizes a balance blank with every batch of samples, consisting of a weighing dish passed through all steps of the procedure.

15.5 *Method verification standard:* A method verification standard should be run with every batch of samples. A solution containing 2% (w/v) sodium chloride can be prepared and used for this purpose, provided it is stored in a tightly sealed container. Process 5.0 mL of this solution in the same manner as a sample.

15.6 *Calibration verification standard:* Not applicable.

15.7 *Definition of a batch:* Any number of samples which are analyzed together and recorded together. Samples within a batch must be of the same matrix. The maximum size of a batch would be limited by the equipment constraints. A batch cannot be larger than what is practical with the equipment.

15.8 *Sample size:* 10 mL sample minimum, 15 mL recommended.

15.9 *Sample storage:* Samples should be refrigerated.

15.10 *Standard storage:* Not applicable.

15.11 *Standard preparation:* Not applicable.

15.12 *Control charts:* The results of the method verification standard are to be control charted.

16. Keywords

16.1 Moisture, total solids, biomass.